6.0 LABORATORY PROCEDURES

The establishment of good laboratory practices is paramount to obtaining quality results from samples collected under the effluent monitoring and environmental surveillance program specified in DOE 5400.5. Laboratory procedures and practices *should** be documented in the site Environmental Monitoring Plan (in compliance with DOE 5400.1) to show

- Sample identification systems
- Cross-contamination prevention measures
- Sample preservation and handling practices
- Analytical methods (standard methods)
- Modifications to any standard analytical methods
- Analytical capabilities (in-house and outside analytical contract capabilities)
- Equipment-calibration and reference-source (check-source) practices (including procedures, frequencies, and methods for tracking/managing)
- Other quality assurance procedures.

6.1 SUMMARY OF LABORATORY PROCEDURE REQUIREMENTS

The summary presents the laboratory measurement procedural requirements necessary for a DOE site. A site does not have to maintain a full laboratory, but it does need to have the necessary laboratory capabilities available to it.

6.1.1 Sample Identification System

Each monitoring and surveillance organization *should** have a sample identification system that provides positive identification of samples and aliquots of samples throughout the analytical process. The system *should** incorporate a method for tracking all pertinent information obtained in the sampling process.

6.1.2 Procedures Preventing Cross-Contamination

To prevent incorrect analysis results caused by the spread of contamination among samples, each laboratory *should** establish and adhere to written procedures to minimize the possibility of cross-contamination between samples. High-activity samples *should** be kept separate from low-activity samples. In addition, the integrity of samples *should** be maintained; that is,

the degradation of samples *should** be minimized by using proper preservation and handling practices that are compatible with the analytical methods used.

6.1.3 Documentation of Methods

To provide that the analyses performed are consistent and of the highest quality, specific analytical methods *should** be identified, documented, and used to identify and quantify all radionuclides in the facility inventory or effluent that contribute 10% or more to the public dose or environmental contamination associated with the site. Standard analytical methods *should** be used for radionuclide analyses (when available), and any modification of a standard method(s) *should** be documented. In addition, methods, requirements, and necessary documentation *should** be specified in any analytical contracts established with outside laboratories.

6.1.4 Gamma-Emitting Radionuclides

All sites that release or could release gamma-emitting radionuclides *should** have the capability (either in-house or outside) of having samples analyzed by gamma-ray spectroscopy systems. This requirement applies to all samples whether they are routine, special, or emergency samples.

6.1.5 Calibration

Counting equipment *should** be calibrated using, at a minimum, the calibration frequency recommendations of the manufacturers so that accurate results are obtained. In addition, check sources *should** be counted periodically on all counters to verify that the counters are giving correct results.

6.2 HANDLING OF SAMPLES

To comply with the sample-identification system requirement, all pertinent information on the samples and their analysis *should* be recorded in a permanent laboratory record book and/or computer system with hardcopy backup. The sample identification number *should* enable tracking of the exact location of the record entry or computer file and indicate the chain of custody for the samples.

6.2.1 Measurement (Screening) of Activity Levels Using Monitoring Equipment

Environmental samples collected in the vicinity of nuclear facilities could have widely ranging levels of radionuclides. They could also have radionuclide contamination in forms and levels that could contaminate materials and equipment with which they come in contact. Therefore, except for control samples or samples that historically have had very little or no activity, such environmental samples *should* be surveyed to determine activity levels and to detect transferable contamination before they are brought into the laboratory. Special precautions, such as the use of lead shielding or extra PVC bags, *should* be taken with samples that show elevated activity levels.

6.2.2 Shipping, Unpacking, and Repackaging of Samples

Samples that are sent offsite for analysis or for laboratory intercomparison *should** be monitored for contamination and radiation levels and packaged in a manner that meets applicable transportation regulations and requirements. Samples that have been prepared with nitric acid may be considered to be hazardous substances and *should* be transported accordingly. Samples that show measurable surface contamination *should* be repackaged in uncontaminated containers before they are brought into the laboratory. This repackaging is necessary to prevent the spread of contamination or the loss of sample constituents. Even samples that do not show measurable surface contamination, using survey instruments, can have activity levels that can result in serious contamination of laboratories and counters. Also, sample containers prepared in the field are often poorly sealed, which can result in portions of the sample leaking out of the container. Therefore, all inadequately packaged samples *should* be repackaged before they are brought into the laboratory. The repackaged samples *should* be packaged in at least double containers to prevent contamination if one of the containers leaks. The outer container *should* be handled only by a person who has had no contact with the sample or other contaminated materials. For example, a water sample can be sealed in a plastic bottle by a person who is believed to be uncontaminated. The bottle can then be placed into a plastic bag held by a person who has had no contact with the sample or other radioactive materials. The plastic bag *should* then be sealed airtight. In cases where the samples could have high levels of radioactivity, it would be prudent to heat-seal the bottle and plastic bag in another plastic bag to help prevent the escape of radioactive materials from the package.

6.2.3 Prevention of Cross-Contamination

High- and low-activity samples *should* be treated in different laboratories, or at least in separate, distinct locations of the laboratory. The measurements made during sample screening with survey instruments *should* be among the criteria used to determine which laboratory (location) will receive the sample. Laboratory glassware that has been used in processing highly radioactive samples *should* be appropriately discarded and not reused. A clean material, such as bench paper, *should* be used to cover laboratory benches before processing a set of samples. Periodic surveys of gross activity levels in the laboratory *should* be conducted to detect any contamination that might occur. Detected contamination *should* be removed by proper decontamination practices. Following physical and chemical treatment of the original samples, the resulting samples *should* again be sealed in plastic bags before being transported to the counting room for counting.

6.2.4 Selection of Sample Sizes According to Gross Beta and Gross Alpha Activities

The size of the sample counted will depend on the activity of the sample. If the activity of the sample is near background levels, it could be necessary to count as large a portion of the sample as is practical for as long as is practical to obtain measurements with the desired degree of sensitivity and

precision. Other samples may produce counting rates that are too high for the counter, producing coincidental readings that are inaccurate. These will produce artificial peaks with energies that are the sum of the energies of other peaks. Very high counting rates can also produce unacceptable counter dead times. In this case, it may be necessary to count only a small (representative) portion of the sample for a short period of time. Gross beta, gross alpha, and gross gamma measurements *should* be used to determine the most suitable sample size.

6.2.5 Preparation of Samples

The chemical separation procedures, if any, that will be necessary to prepare samples for counting will depend on the nature of the sample and the radiation emitted by the radionuclide of interest. Radionuclides that emit gamma radiation will generally not require chemical separations, but alpha or beta emitters generally will. Chemical separations *should* be avoided whenever possible because of the time and expense involved and because of the errors that can result from radionuclide losses during chemical separations. Carriers and/or tracers *should* be introduced at an early stage of any procedure requiring chemical separations under conditions that will maximize isotopic exchange so that chemical yields can be calculated. The following subsections present the general types of separation procedures that might be required for different types of samples.

6.2.5.1 Air

Atmospheric concentrations of radionuclides attached to (or in the matrix of) aerosol particles *should* be measured by directly counting air-filter samples using low-background detector systems without any chemical separation. Photon emitters *should* be measured directly using germanium diodes without chemical separation. Chemical separations *should* be used only in cases where the concentrations or the photon energies are very low. If the particulate material is collected on the filter surface, the deposit does not become too thick, and interfering radionuclides are not present, then concentrations of alpha emitters *should* be measured directly from an air filter using alpha spectrometers. Samples collected using membrane filters *should* be counted directly for alpha emitters because membrane filters collect particles on the surface. However, the air flow rate that is possible through membrane filters is much less than that through fibrous filters, which causes the membrane filter to plug more rapidly. Therefore, alpha emitters that are present in low concentrations in the atmosphere often cannot be detected using membrane filters. Samples containing low concentrations of alpha emitters *should* be collected at high flow rates on fibrous filters and chemically separated before counting. High concentrations of naturally occurring short-lived radon and thoron decay products on air-filter samples can seriously affect the measurement of other radionuclides. The concentrations of the thoron decay products are generally 1 to 3 orders of magnitude lower than those of radon decay products. The short-lived radon decay products decay with an effective half-life of about 30 minutes, and the thoron decay products decay with a half-life of about 11 hours. Therefore, air-filter samples *should* be allowed to stand several hours before counting to allow the radon decay products to

decay, or several days to allow both radon and thoron decay products to decay, rather than chemically separating the radon and thoron decay products. Many radionuclides in the atmosphere are in the gaseous phase and are not attached to (or in the matrix of) aerosol particles. These radionuclides are measured in whole air samples, in samples collected in cold traps, or in materials that have been used to chemically or physically absorb the radionuclides from the air. Unless the concentrations are too low, photon-emitting radionuclides collected on absorbent materials can be measured directly without chemical separation. Alpha and beta emitters generally require chemical separations. Noble gases are usually present in the gaseous effluents of nuclear facilities, such as nuclear reactors and fuel reprocessing plants, and are typically measured in whole air samples. For reactors, the shorter-lived radionuclides such as ^{41}Ar, ^{133}Xe, and ^{135}Xe will usually be the most important. Irradiated fuels are typically stored 6 months or more before reprocessing, so only the longer-lived nuclides, such as ^{85}Kr, are expected to be found in the environment around a reprocessing plant. Krypton-85, ^{41}Ar, ^{133}Xe, and ^{135}Xe are measured by gamma counting. For facilities involving ^{226}Ra or thorium, the release of ^{222}Rn or ^{220}Rn will need to be considered.

6.2.5.2 Water

A major concern in the measurement of radionuclides in water is the preservation of the samples before counting, especially if the distribution of radionuclides between an aqueous and a solid phase is desired. Continuing chemical and biological action in the samples can cause changes in the chemical and physical form, deposition on the container walls, and removal of the radionuclides to biological growths. Phenomena that *should* be considered include

1) Ion exchange of cations between the sample and the container walls (cesium, for example, can exchange with potassium in glass)

2) The absorption of radionuclides by algae or slime growths on container walls or particulate materials

3) The hydrolysis and resulting sorption of radionuclides on container walls or particulates (this is especially likely at the low acidities typical of natural waters and some process streams)

4) The formation of large flocculent particles from radiocolloids resulting in additional plate-out

5) Change in the distribution of radionuclides between aqueous and solid phases as a result of sample pretreatment (e.g., acidification leaching radionuclides from suspended particles)

6) The conversion of iodides to iodine by biocides, followed by the loss of iodine by vaporization

7) The quenching of liquid scintillation cocktails by acids

8) The change of counter geometries by the settling of particles or by their fixation on container walls.

The report EPA 625/6-64-003 lists various preservation methods and permissible storage times for water samples according to chemical species. Current practice at most nuclear installations is to predose the sample container with an acid [typically 2 to 3 mL concentrated H_2SO_4 or HNO_3 (depending on compatibility with subsequent chemistry) per liter of sample], to inhibit biological growth and plate-out of dissolved ions on the container wall. Pretreatment of the sample container with a salt solution of the same chemical species as the radionuclide to be measured can help minimize wall adsorption. Keeping the sample container refrigerated and shielded from light inhibits biological growth. Filtration during sample collection can be effective for some situations. The radioanalytical procedures to be used and the purpose of the measurements *should* govern what, if any, pretreatment is used, because the procedures can be adversely affected by additives used to preserve other radionuclides. Optimum preservation procedures *should* be determined by local testing. The concentrations of gamma-emitting radionuclides in whole water samples *should* be measured directly by gamma-ray spectrometry, if such concentrations are high enough for determination. For accurate measurements, the radionuclide distribution *should* be uniform throughout the sample. If solids settle out of the sample, the geometry of the sample is changed, which makes it necessary to filter the water and count both the filter and the filtered water. If the distribution of the radionuclides between the solid and the aqueous phases is desired, the water sample *should* be filtered during or as soon as possible after collection, before acidification, and the water and filter counted separately. If additional precipitate develops later, the water *should* be filtered again just before counting. However, the precipitate in this case *should* still be considered to be part of the liquid phase. If concentrations of gamma emitters are too low to be measured in the whole sample, the sample *should* be concentrated by evaporation or placed in a 2-Pi counting configuration to maximize detector efficiency. If the concentrations are still too low to be measured in an evaporated sample, or if beta or alpha emitters are to be measured, the radionuclides to be measured *should* be chemically separated using procedures that will be determined by the radionuclides required.

6.2.5.3 Soil and Sediments

Since the water content of samples can vary widely, soil and sediment samples *should* be dried according to procedures that have been established for the measurement program, and the measured radionuclide concentrations reported on a dry-weight basis. Oven-drying temperatures ranging from 80°C to 130°C can be used; however, a fixed temperature, such as 110°C, *should* be used for all samples. The oven temperature *should* be set according to the substance being analyzed for; e.g., use an oven temperature of 100-105°C for samples containing volatile organic compounds. Freeze-drying (drying under vacuum) is an excellent but expensive alternate method for drying samples. It is especially useful for large samples that contain considerable organic matter, which could undergo combustion during oven-drying. The loss of radionuclides by volatilization and by frothing and spattering during drying is also

minimized by freeze-drying. Soil and sediment samples can be counted directly for some gamma-emitting radionuclides if the concentrations are high enough. However, to obtain accurate results, the samples *should* be homogeneous. So that soil samples are homogeneous, they *should* be ground to a small particle size and homogenized before counting. To determine the particle size distribution of the radionuclides, sieves can be used to separate the original sample into particle-size fractions. Small rocks and pebbles *should* be separated from the sample before counting. Radionuclides of interest in soil and sediment samples *should* be chemically separated where necessary to obtain the desired sensitivity. High concentrations of gamma-emitting radon and thoron decay products in soil can interfere with the measurement of low concentrations of other gamma-emitting radionuclides. Alpha and beta emitters cannot be measured directly, unless they are present in high concentrations, because of the short range of the alpha and beta particles and the high concentrations of alpha and beta emitters in the uranium and thorium decay chains.

6.2.5.4 Biological Materials

In some cases gamma-ray spectrometers can be used to measure gamma-emitting radionuclides in biological samples without performing chemical separations. Where appropriate, freeze-drying can be used to decrease the weight of the sample. However, when large amounts of biological material are present, wet- or dry-ashing and chemical separations *should* be performed before counting the samples, especially in the case of alpha- or beta-emitting radionuclides. The choice of whether to wet- or dry-ash a sample is dependent on its properties, such as mass, bulk, physical form, oxidation resistance, and volatility of the desired constituents. Dry-ashing is simpler but could result in the loss of elements that are volatile at ashing temperatures. Also, refractory residues can form, and part of the desired material could even combine with the container. Porcelain, silica, nickel, and platinum all have an affinity for certain elements at ashing temperatures. These problems can be minimized by ashing at lower temperatures, such as 400°C to 450°C, but this prolongs the ashing process. Also, many samples can ignite, producing local temperatures that are far in excess of furnace temperatures (HASL-300). Wet-ashing is more tedious, particularly for large samples, but volatilization during wet-ashing will occur only with extremely volatile elements such as iodine or bromine. Therefore, wet-ashing is preferable when there is no direct evidence that dry-ashing is suitable for the particular sample. Wet-ashing also has the advantage that carriers can be added directly during the ashing process (HASL-300). The major oxidizing agent used is nitric acid, and frequently the complete oxidation can be carried out with this agent alone. The addition of sulfuric or perchloric acid to assist oxidation is sometimes useful, but it can lead to the formation of insoluble compounds such as barium sulfate, calcium sulfates, or potassium perchlorate. In addition, high temperatures are reached when these acids are evaporated, which can lead to increased volatilization loss. Kjeldahl treatment can provide rapid ashing in cases where the added sulfuric acid does not present a problem (HASL-300).

6.2.5.5 Sample Preservation

It is essential to maintain the integrity of samples (i.e., to minimize degradation of samples by using proper preservation and handling practices that are compatible with analytical methods). Degradable biological materials *should* be kept frozen until they are processed. A small amount of acid *should* generally be added to water samples to inhibit biological growth and the plate-out of dissolved materials on the container walls. However, acid *should* not be added in cases where the sample contains radionuclides that are volatile in acid solutions. A reducing agent, such as Na_2SO_3, *should* be added to solutions containing ^{129}I or ^{131}I to prevent the formation and loss of I_2. Refrigeration, shielding from light, and filtration *should* be used when necessary to prevent biological growth and deposition on container walls.

6.2.6 Sample Archiving

Sample archiving refers to the storage of samples for a period longer than is normally required to perform the routine sample analysis and result verification. Samples may be archived either before or after sample preparation and analysis. Routine sample analysis and result verification *should* normally be completed within 90 days of collection. However, special conditions might exist any time that routine sample analysis activities are disrupted. In these cases, it may be necessary to consider the factors listed below even for routine samples.

Decisions to archive environmental samples *should* be based on an identified future need for the sample. The decision to archive samples *should* be documented and re-evaluated on an annual basis for archive periods greater than one year.

For most cases, long-term archiving may not be required. However, special samples (e.g., those associated with accidents or those obtained to respond to public concerns) might be candidates for archiving. For periods when routine analysis activities may be interrupted or otherwise incapable of providing analysis results, the need for short-term archiving (i.e., months to a few years) of representative samples from routine environmental surveillance *should* be considered. The need for archiving special samples for longer periods (i.e., tens of years) *should* also be addressed.

The following factors *should* be considered when making a decision to archive samples:

1) Suitability of analyte - Determine the suitability of the radionuclide for archiving. For example, short-lived nuclides can be stored for only a short time before radioactive decay makes the sample unusable for analysis. The minimum detection limit of the analytical methods should be considered. Radionuclides that are in a volatile physical form, such as organics, also may not be appropriate for archiving. These factors *should* be considered in conjunction with the archive period expected. For example, the archiving of charcoal filters for analysis of I-131

(8-day half-life) would be inappropriate, in contrast to archiving them for analysis for I-129 (1.6×10^7-year half-life).

2) Media compatibility - Determine whether the medium can be archived and for what period of time. For example, milk can be very difficult to store, as it spoils on the shelf and thickens when frozen and thawed. Normally, liquid samples are not suitable for archiving over long periods. Consequently they *should* normally be retained for short periods only. In most cases, only solid samples or filters can be archived for extended periods. These types of samples are generally ashed or require no special treatment prior to analysis, and media compatibility is less of a concern.

3) Special sample preparation for storage - Prior to archiving, special sample preparation that is different from that normally used in preparation for analysis may be required. It may be necessary to partition the sample before archiving for subsequent evaluation of different radionuclides. For example, water samples may be acidified to prevent algal growth or plateout of particulate radionuclides. However, acidification may cause the loss of any tritium and radioiodines present. Vegetation may be carefully dried to prevent decay; however, volatile substances may be lost during drying. It may be necessary to place heavily loaded air filters on metal planchets inside Petri dishes to help prevent dust loss during handling and storage.

4) Type of container - Consideration *should* be given to the suitability of the container for long-term storage. Nuclides may tend to plate out or be absorbed into the walls of some types of containers. Containers must not degrade during the expected archive period and *should* be resistant to attack from insects and mice, the problem of mice being of particular concern for plastic storage bags. Containers may be required to prevent light from reaching and degrading the sample, or double containment may be necessary to guard against breakage and loss of sample or spread of contamination.

5) Sample analysis - The type of analysis performed on a sample that has been archived may be quite different from that performed on fresh samples, and special laboratory procedures may be needed. For example, particulates may settle out of liquids that have been stored for long periods and may have to be resuspended. It may be necessary to rinse the planchet holding heavily-loaded air filters with nitric acid to collect dust shaken loose from the filter. Analysis of milk may normally be done by passing it through a resin column; however, analysis of an archived, thickened product would necessarily be quite different, and the difference may limit the types of radionuclides that could be analyzed for. The possible ingrowth of radioactive decay products should be considered.

6) Quality assurance - Ensure that samples are properly logged and stored, and that sample accountability is maintained and documented. Maintaining sample accountability is critical in determining the future usefulness of

the sample, regardless of sample storage or analysis capabilities. Sample archiving *should* be addressed in the Quality Assurance Plan associated with the facility.

7) Storage capability - The quantity of shelf space, freezer space, or special storage needed, as well as light or darkness requirements, *should* be determined based on the period over which samples are to be collected and archived. The need for physical security and restricted access *should* also be considered.

8) Impact on routine program - For ongoing analysis programs, consider the impact that future analysis of archived samples will have on the capacity for routine analyses under way in the future. Analyzing archived samples may adversely impact future routine analyses of samples by overloading laboratory capacity.

9) Data compatibility - Data obtained from archived samples *should* be compatible with and comparable to existing data. Any proposed change in analytical techniques or data analysis methods *should* be evaluated and their effect determined before they replace current methods on actual samples or sample data. A side-by-side comparison of the current and proposed methods on sample aliquots or duplicates *should* be considered.

10) Sample disposal - Determine the possible impact of disposal of samples that have been archived but not analyzed. Consider whether the samples will be disposed of as low-level radioactive waste, hazardous waste, or mixed waste, and any special disposal or storage requirements under RCRA.

6.3 ANALYSIS METHOD AND CAPABILITIES

Excellent references for analytical methods are APHA (1977, 1985), IDO-12096, EMSL-LV-0539-17, EPA-R4-73-014, EPA-600/4-80-032, EPA-520/5-84-006, and HASL-300. Drinking-water samples *should* be analyzed using EPA procedures where such methods are available and adequate for the radionuclides of interest. Alternate methods can be used in cases where satisfactory EPA-approved methods are either not available or not adequate. However, such alternate methods *should* have documented or documentable evidence showing that they give reliable results.

6.4 GROSS ALPHA, BETA, AND GAMMA MEASUREMENTS

Gross alpha and beta measurements *should* not be used to characterize a sample. Sample characterization *should* be done using radionuclide-specific analyses. However, gross alpha and beta measurements can be useful in determining the general activity level of the sample so that proper choices can be made regarding the size of the sample and the appropriate chemical separation procedures. Gross alpha and gross beta measurements *should* be made using a gas-proportional counter. Gross gamma measurements *should* be made using gamma-ray spectrometers.

6.5 DIRECT GAMMA-RAY SPECTROMETRY

Gamma rays *should* be measured directly using sodium iodide thallium activated crystals [NaI(Tl)], lithium-drifted germanium diodes [Ge(Li)] or hyper-pure germanium type detectors (HPGE). The energy resolution of NaI(Tl) crystals is much poorer and the background is much higher than those of germanium diodes, which severely limits the number of radionuclides that can be measured in complex mixtures using NaI(Tl) crystals. However, NaI(Tl) detectors are still useful on samples that have relatively simple spectra or on radiochemically separated samples. For low-energy photons, IG diodes are somewhat more efficient than Ge(Li) diodes.

6.6 BETA COUNTERS

Beta-emitting radionuclides *should* be measured using ionization, gas-proportional, or liquid scintillation counters. Carbon-14 is often converted to a gas, such as CO_2 or CH_4, which is used as the counter gas during counting. Most beta emitters are counted with the sample outside the counter. A commonly used counter consists of a hemispherical chamber with a window on the flat end. The counter window can be covered with a thin polyester film. If the window is not covered with a polyester film, the sample holder must be attached to the counter in such a manner as to prevent the escape of gas through the window. In a liquid-scintillation counter, the sample is dissolved in scintillating liquid and placed in a standard-sized vial. Beta particles impinging upon the scintillating liquid in the vial produce light flashes that are measured using photomultiplier tubes.

6.7 ALPHA-ENERGY ANALYSIS

High-resolution alpha spectrometry using silicon surface barrier detectors *should* be used to determine the concentrations of alpha-emitting radionuclides in thin, uniform samples or in samples that can be deposited as thin, uniform sources. The accuracy and sensitivity of the measurements decrease considerably with increasing sample thickness because the matrix absorbs and scatters alpha particles. Therefore, chemical separations followed by the formation of thin deposits are necessary for more massive samples. Chemical separations are also necessary to resolve radionuclides that emit alpha particles with energies that differ by less than about 50 keV. Electrodeposition is the method that *should* be used to produce thin, uniform sources. However, the wide range of environmental and biological samples makes it difficult to develop electrodeposition procedures that can handle all types of samples. A coprecipitation method using rare earth compounds, such as neodymium or lanthanum fluoride, to separate actinides can provide a sample mount that in many cases is equivalent to an electrodeposited sample. Alpha spectrometry *should* be used primarily for the analysis of actinide radionuclides because the concentrations of these radionuclides in environmental samples are often near the detection limits of the alpha spectrometer, and because large samples are often needed to produce detectable counting rates. Therefore, very efficient separation procedures are needed to decrease the concentrations of

impurities in the deposited samples. Most deposition procedures are very sensitive to hydrolytic losses; even microgram quantities of impurities can cause problems with yield and resolution.

6.8 RADIOCHEMICAL SEPARATION PROCEDURES

Innumerable radiochemical separation techniques have been used by various investigators to separate the radionuclides being evaluated from interfering radionuclides. No general set of separation procedures can be specified that will apply to all conditions at all DOE sites. However, standard (professionally accepted) methods *should* be used when separating radionuclides from interfering radionuclides.

6.9 REPORTING OF RESULTS

The reported analytical results *should* include the 2σ uncertainty limits. The reported uncertainty limits *should* be calculated from the statistical counting error and as many other sources of error as can be identified. Each random error *should* be reported separately. The concentrations *should* be reported as calculated even when they are less than the error limits or negative, because such concentrations are required for the statistical analysis of the data. Values that are negative or below detection limits *should* be reported using a symbol and stating, in a footnote to the table, that the value is below the lower limit of detection. In all cases, the error limit *should* be given so that a detection limit can be inferred. The results for short-lived radionuclides *should* be decay-corrected to the midpoint of the sample-collection interval.

6.10 COUNTER CALIBRATION

Proper and timely calibration of counting equipment is essential if accurate analytical results are to be obtained. Except in gamma-ray spectrometry when NIST-traceable standards are used to prepare counting efficiency curves, each counter *should* be calibrated for each radionuclide to be measured using standards traceable to the NIST. The standard *should* have the same geometry and matrix as the sample to be counted, and the standard *should* be well-mixed and remain well-mixed throughout the matrix that is used to produce the standard geometry. Many different procedures have been used to produce standards of different shapes and sizes. A recommended procedure for calibrating a gamma detector for solid samples is one in which the standard is pipetted onto Al_2O_3 powder. After the standard has dried, the Al_2O_3 powder is mixed thoroughly. The powder is then mixed thoroughly with an epoxy resin, which later solidifies to produce a solid that is very resistant to breakage and will not allow the standard to migrate. If a gamma counter is calibrated for several radionuclides, a plot of efficiency versus energy *should* be prepared and used to identify errors in the calibration of individual radionuclides and to determine the efficiencies of radionuclides for which standards are not available.

6.11 INTERCALIBRATION OF EQUIPMENT AND PROCEDURES

Interlaboratory exchanges of samples *should* be carried out to determine whether the laboratories are obtaining the same results, and to eliminate any problems that are causing discrepancies. If samples are available that have not been chemically separated but are still known to be homogeneous, aliquots of these samples *should* be exchanged so that both the separation procedures and the counting equipment can be compared.

6.12 COUNTER BACKGROUND

One of the major factors that determines the sensitivity of the measurement procedures is the background of the counter. Therefore, the counter background *should* be reduced as much as possible. The counter *should* be shielded with lead or other materials, such as borated paraffin (to absorb neutrons). However, lead shielding will not significantly reduce the background caused by high-energy cosmic rays. The background from cosmic rays can be reduced by surrounding the sample counter with an anticoincidence counter(s). When primary cosmic rays interact with atmospheric gases, they produce showers of secondary cosmic rays that will produce simultaneous counts in the sample counter and the anticoincidence counter(s). Radiation that is emitted by the sample generally will not produce pulses in both the anticoincidence and the sample counters. The pulses in the sample counter that are simultaneous with pulses in the anticoincidence counters are then automatically rejected. The background of the counter *should* be kept low by preventing the contamination of the counter by radioactive materials. Such contamination not only would raise the background, but also would result in spurious measurements. Therefore, backgrounds *should* be measured regularly, and the counter decontaminated if background measurement shows evidence of contamination.

6.13 QUALITY ASSURANCE

As they apply to laboratory procedures, the general quality assurance program provisions of Chapter 10 *should** be followed. Specific quality assurance activity requirements for laboratory operations at a site *should* be incorporated in the facility's plan for quality assurance.